INVESTIGATION ON WATER HYACINTH (EICHHORNIA CRASSIPES) NATURAL PLANT BASED POLYMER COMPOSITE MATERIAL PROPERTIES

A SYNOPSIS

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1. INTRODUCTION

In composite materials, two materials are combined with different physical and chemical properties. The combination of these elements creates a material that can be specialized to perform certain tasks, such as becoming stronger, lighter, or less susceptible to electrical power. The material can also be made stronger and stiffer. As opposed to traditional materials, they are preferred because they have improved properties and are applicable to a wide range of uses. The individual constituent materials do not combine to form a composite material, however, the large range of availability of the secondary and primary reinforcement materials allows the designer to choose the optimal combination based on the application. The natural world contains composites. Wood is a composite made of cellulose fibres and lignin, which hold them together. Composite material's unique properties come from its combination of materials. Composite materials have been used by humans since thousands of years ago in a variety of applications.

Water hyacinths (Eichhornia crassipes) are free-floating aquatic plants that are mostly found in tropical and subtropical regions. Their special characteristic is their rapid growth rate. There are enough plants to cover the entire surface of the water bodies with ten to twenty plants. Plants growing in water bodies usually reach a height of five or eight centimetres. Compared to other natural plants and fibres these plants have very fast-growing characteristics and are able to withstand 28 years on the water surface. As a result, water bodies become less oxygenated and nutrient-filled. It is for this reason that other plants and animals were also killed by this water hyacinth. The plants possessed hydrophilic properties. They absorbed 70 percent of the water they consumed. Various applications and ornamental items were made from the separate parts of Eichhornia crassipes, such as the stem, petioles, and roots. High problematic plants are simply referred to as that. The stalks are long. One or two daughter plants are produced by each water hyacinth plant. They are more resilient and can survive for a longer period. It is reported that some water hyacinth plants reach a height of 2 to 5 meters in a particular region, and in some regions, these plants reach up to 8 m.

Historically, natural fibre composites (NFCs) have been used since ancient Egyptian times, when they made bricks from clay, mud, and straw. During the past few years, natural fibres as reinforcement for polymer composites have gained a lot of attention, especially in the research community, and there have been numerous reviews of NFCs in the literature. In recent years, NFCs have mostly been used in wood-plastic composites and automotive inner door panels. As compared to synthetic fibres, natural fibres offer advantages in terms of the environment, economy, and technical properties. Some species of natural fibre can be harvested up to six times a year, not just renewable but rapidly renewable as well. Sustainable aspects of plant fibres are becoming more widely known as public awareness about them increases, and this will likely lead to a greater interest in their commercial use.

Due to the rapid growth of these plants, the conductivity, dissolved oxygen, TDS, and salinity of water were all decreased. Cellulose, hemicellulose, and lignin have usually been found in lignocellulose compounds. In some of the pre-treatment experiments, the cellulose, hemicellulose, and lignin contents of these plants were separated. Various types of pre-treatments have been implemented. Some fractionation, solubilisation, and hydrolysis take place the same way. Ecosystems were severely harmed by these plants. Water irrigation, tourism, and fisheries departments are at risk because of it. With the help of daughter plants and by their thick mats and fastest-growing characteristics, within one month this plant was fully covered all over the surface of water bodies. Hyacinth plants normally grow only in water bodies, so they contain more cellulose than other aquatic plants. Water hyacinth growth is also affected by local temperature. Generally, the hyacinth plant grows best at temperatures between 20°C and 26°C, 25°C to 32°C is the ideal temperature, and above 35°C is the highest temperature.

Some of the worst plants in the world are listed by the International Union for Conservation of Nature. According to IUCN, water hyacinth was one of the top ten plants that were considered to be hazardous in aquatic environments. In India, this hyacinth plant was also considered trash by the governments. Basically, the waste management concept is easily adapted to the removal and proper use of hyacinth plants.

2. LITERATURE REVIEW

This chapter elaborately discusses the earlier works done in the natural fibre reinforced polymer composite fields. And, also these literature reviews are fulfilling the essential information on natural fibre composites for our present needs regards sustainable environments. This chapter explained the different research areas related to polymer matrix composites include the mechanical (tensile, flexural, impact), characterization, thermal behaviour, both water and chemical absorption, and filler materials. This section deeply analyses the research works done by the previous researchers and previous kinds of publications for the following field:

Pietak et al. [1] examined hemp fibre wettability and atomic force characterization. From these experiments, the raw hemp fibre is treated the steam alkaline, combinative steam alkaline, and enzymatic treatments. These treatments increase the wettability and improve the adhesion behaviour of the atomic force microscopy tip and substrate.

Fiore et al. [2] investigated the effect of sodium bicarbonate chemical treatments on the sisal fibre reinforced epoxy polymer composites. The sodium bicarbonate chemical treatments are done at different hours to the sisal fibre. Finally, it improves the interfacial adhesion to the sisal fibre and epoxy polymer matrix as well as improves the tensile strength of the sisal fibre reinforced polymer composite samples.

Chandrasekar et al. [3] reviewed the effect of the different types of chemical treatments done to natural fibres such as alkalization, benzoylation and acetylation. These various treatments have highly improved the bonding behaviour of the natural fibre reinforcement to the matrix material phase. And also, these treatments are reduced to the water absorption characteristics to the fibre composite sample.

Cruz and Fanguerio [4] reviewed the different types of surface modifications like chemical treatments cone by the natural fibres such as alkali treatment, silane treatments, and used to some water-repelling agents. This technique improves the mechanical properties of the natural fibre composite sample and modifies the crystal structure of the fibres.

Agnivesh et al. [5] investigate the effect of alkali treatments on abaca natural fibre. This work abaca fibre is treated with NaOH solution at different weight percentages. It improves the bonding behaviour of the reinforcement fibre and the matrix material. The improvement of interfacial adhesion is examined by using a scanning electron microscope. Yahaya et al. [6] examined the NaOH chemical treatment process on kenaf aramid hybrid laminated composites. In this work, the kenaf fibre mat is treated with different types of sodium hydroxide (NaOH) solutions. Then, the treated and untreated hybrid composites are compared. Based on the final experiment results, chemically treated kenaf aramid hybrid composites have achieved higher mechanical strength.

Asim et al. [7] studies the effect of alkali and silane treatments to the kenaf pineapple fibres. The alkali, silane and combination of alkali-silane treatments are improved the mechanical properties of the composite samples.

Syafri et al. [8] characterize the microfibers from the water hyacinth plant and utilize those fibres as a filler material to the sago starch bio composites. These microfibers are varied with different weight percentages like 0, 5, 10, 15, and 20%. Based on the experiment results the 15% water hyacinth cellulose fibre with sago starch composite sample is attained high tensile strength of 10.23MPa. The mechanical failures and fibres pull-out natures are examined by using a scanning electron microscope.

Abral et al. [9] investigated the alkali treatment effects on the water hyacinth plant fibre. These plant fibre are reinforced with unsaturated polyester composites. In this research the hyacinth plant fibres are extracted from the retting process and the parent plant stem. The different concentration of alkali treatments is done (2, 5, 7m, 10%) by the hyacinth plant fibres. The volume fractions of the hyacinth fibre is maintained at 20% constant. The 7% alkali-treated composite samples attained high mechanical strength like flexural modulus 110.3MPa, flexural modulus 4.7GPa, tensile strength 15.4MPa, and tensile modulus 3.5GPa. Increasing the alkali treatment percentages affect the mechanical properties of the composite samples.

Abral et al. [10] discussed the water hyacinth plant fibre reinforced with polyester composites' mechanical properties. In this work the composite mechanical

properties are evaluated by two methods like before immersion and after immersion in water. With the immersion of water, composite samples have lower tensile and flexural strength compared to the without immersion of hyacinth fibre composite samples. The without immersion composite samples attained 25MPa tensile strength with 2340MPa tensile modulus and 62MPa flexural strength with 1400MPa flexural modulus.

Supri et al. [11] discussed the effect of treated and untreated water hyacinth fibre reinforced with low-density polyethylene composite samples on mechanical, absorption, and morphological behaviour. Based on this study, increasing the hyacinth fibre reinforcement after a particular point affects the mechanical strength of the composite samples. Especially, the tensile strength of the composite sample is decreased. But, from the earlier stages adding the fibre contents, increases the tensile strength of the composite samples. The water intake from the absorption tests depends on the cellulose contents. The untreated hyacinth fibre has absorbed more quantity of water compared to the treated hyacinth fibre composite samples.

Ganesh and Anand [12] reported the staking sequence effect on the tensile properties of hybrid natural fibre composites. The kenaf and basalt fibre are used in this work. These two fibres are stalked in two different ways. In a first stacking sequence laminate [A] is respectively kenaf-basalt-kenaf-basalt-kenaf fibres are reinforced. In a second staking sequence [B] basalt-kenaf-basalt-kenaf-basalt. Based on the two staking hybrid composites the [B] laminate achieved higher tensile strength 59.2MPa compared to the laminate A (56.2MPa) composite. It shows the basalt fibre core and skin layers are properly bonded and achieve high strength, producing good interfacial bonding compared to the kenaf fibres.

Rahman and Putra [13] tested the tensile properties of natural fibre and synthetic fibre reinforced polymer composites. In this test, the different weight percentages of kenaf, jute, and flax natural fibres, as well as different weight ratios of glass, carbon, and aramid fire, reinforced polymer composite samples are used. The results of this work show that synthetic fibre composites attained high mechanical strength compared to natural fibre reinforced composites. But, the consideration of the sustainable environment, cost, and available aspects the natural fibre composites are mostly recommended.

3. RESEARCH GAP

- Based on our present environmental condition there is a need to utilizing the biological waste of aquatic water hyacinth (Eichhornia crassipes) plants.
- Some of the previous kinds of studies are reported on water hyacinth fibre reinforced composites. Very few of the works are done previously in hyacinth ash-based composites. Till now, no work has to be done by the hyacinth powder reinforced composites.
- Lot of work has been done previously based on eggshell filler materials combined with water hyacinth fibre in a hybrid manner. Very few of the works are reported the hyacinth fibre reinforcement alone with the filler materials.
- These above reasons are the main lead for this present research work.

4. OBJECTIVE

The following objectives were derived from the research gap.

- To fabricate the water hyacinth natural plant fibre reinforced polymer composites.
- To convert the waste water hyacinth plant parts to the fibre, powder and ash particle reinforced polymer composites.
- To compare the mechanical properties of hyacinth composites and other natural fibre composites.
- To observe the absorption behaviour of different types of water and chemical absorption tests.
- To understand the thermal behaviour of hyacinth plant based composite.
- To investigate the egg-shell filler material combined hyacinth plant composites mechanical, thermal properties and characterization studies.

5. METHODOLOGY

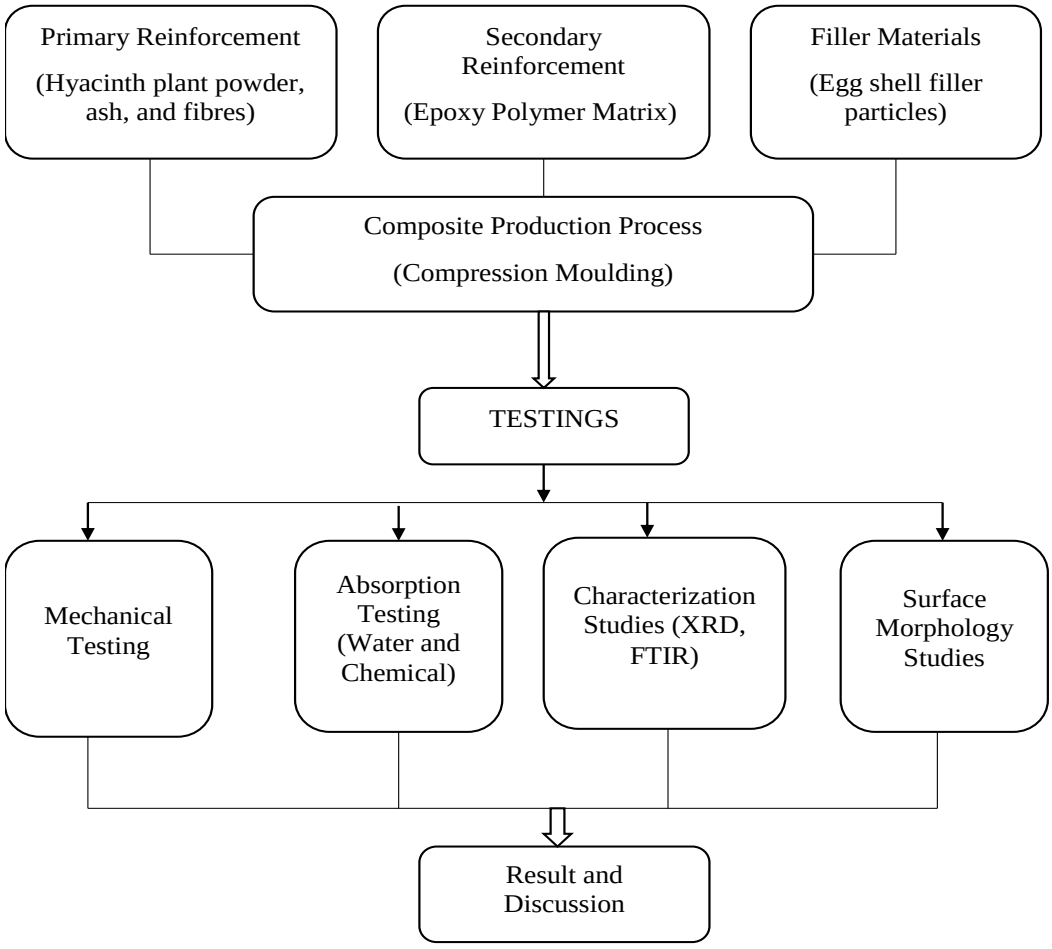

Figure 1 Research Methodology for Hyacinth Plant Based Polymer Composite

6. RESULT AND DISCUSSION

6.1 Mechanical Properties

The above Figure 1 explains the research methodology of hyacinth plant based composite materials. The mechanical strength (tensile, flexural, and impact) of the WH-epoxy composites with respect to different fibre lengths and loadings are presented in Figure 2. The tensile strength of the composites varied from 18.16 MPa to 25.124 MPa with 10 mm WH fibre, 22.42 MPa to 36.16 MPa with 15 mm WH fibre, 34.782 MPa to 42.612 MPa with 20 mm WH fibre, and 32.986 MPa to 36.141 MPa with 25mm WH fibre. The flexural strength of the composites varied from 38.64 MPa to 46.614 MPa, 36.17 MPa to 44.81 MPa, 48.82 MPa to 58 MPa, 44.264 MPa to 48.87MPa for the fibre lengths of 10 mm, 15 mm, 20 mm, 25 mm in the composites.

Initially, the mechanical strength increased gradually for all three types tests. Compared to the different lengths of the fibres with different weight percentages, it was clearly observed that the 30 wt.% of WH reinforcement combined with the epoxy matrix achieved higher mechanical strength compared to the samples with other fibre contents. Further increase in the fibre content (35 wt.%) led to a decrease in the mechanical strength. This trend was observed commonly in all three mechanical tests. This could be due to the fact that at higher fibre content the saturation level exceeded and debonding of the primary and secondary phases started to happen. Furthermore, the higher fibre content created agglomeration effect in the matrix material. It should be noticed that, the flexural strength of 15 mm composite sample was lower than the 10 mm hyacinth composite sample. The processing methods of the primary and secondary reinforcements or manual mixing method could be attributed for this reduction in strength.

In general, the strength of the fibre composites increased with the fibre length up to 20 mm but decreased with a further increase in the fibre length (25 mm) possibly due to the agglomeration effect from the primary fibre reinforcements. Based upon the final strength results, it could be concluded that the 20 mm fibre length with 30 wt.% fibre reinforcement attained higher mechanical strength (tensile, flexural, and impact). From the initial stage of work, sisal, and coir based composite (30% fibre reinforcement, 70% of epoxy polymer matrix) samples were prepared. The sisal composite attained 28.42 MPa tensile strength, 41.24 MPa flexural strength, and 0.25 J impact strength. On the other hand, the coir composite attained 32.628 MPa tensile strength, 41.26MPa flexural strength, and 0.5J impact strength. Compared to the sisal- and coir-based composites, the hyacinth based composite samples attained higher mechanical strength. The pure epoxy resin attained 14.3MPa tensile, 22.14MPa flexural, and 0.20J impact strength. Comparing the mechanical strength of pure epoxy polymer matrix this hyacinth based composites attained high strength values.

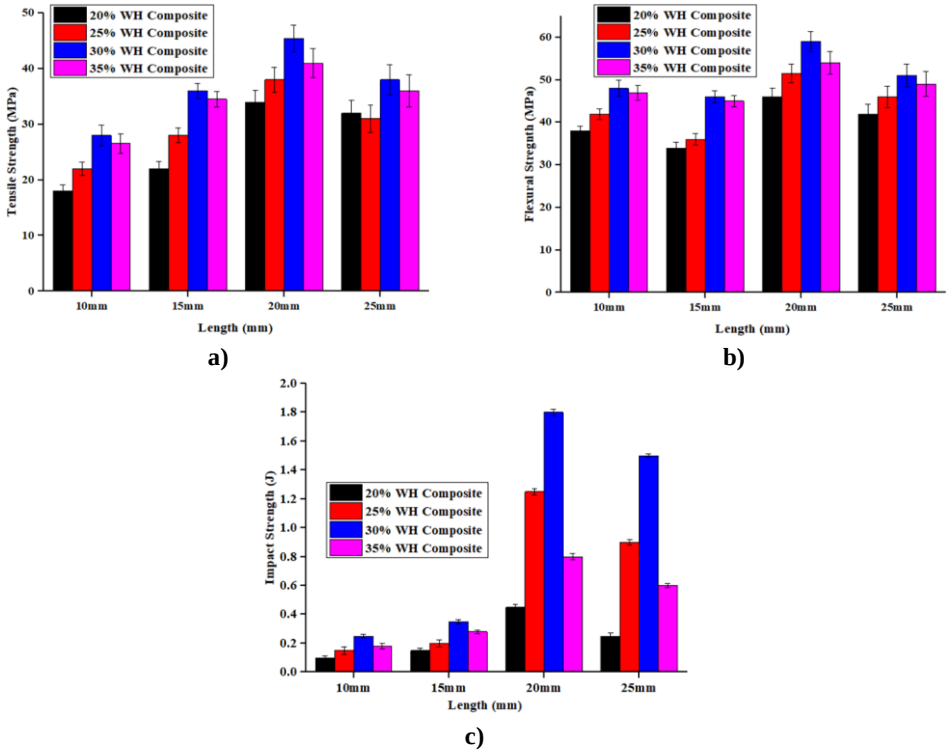

Figure 2 Mechanical strength of WH composites fabricated with different fibre lengths and loadings (wt.%); a) Tensile strength, b) Flexural strength, c) Impact strength

6.2 X-ray Diffraction Techniques

The crystallinity index of WH composites with the 10 mm, 15 mm, 20 mm, and 25 mm of fibre lengths and 30 wt.% fibre content were 48.41%, 51.12%, 60.68%, and 56.27% respectively. Based upon the results, it was clearly understood that the 20 mm fibre length composite showed the highest crystallinity index due to good fibre and epoxy matrix bonding compared to the other samples leading to an increase in the mechanical properties of the composite. However, a decrease in CI with fibre length greater than 20 mm indicated poor interfacial bonding and resulted in lower mechanical strength. The below Figure 3 and Table 1 clearly explains the crystallinity peaks and index of hyacinth plant fibre reinforced polymer composite materials.

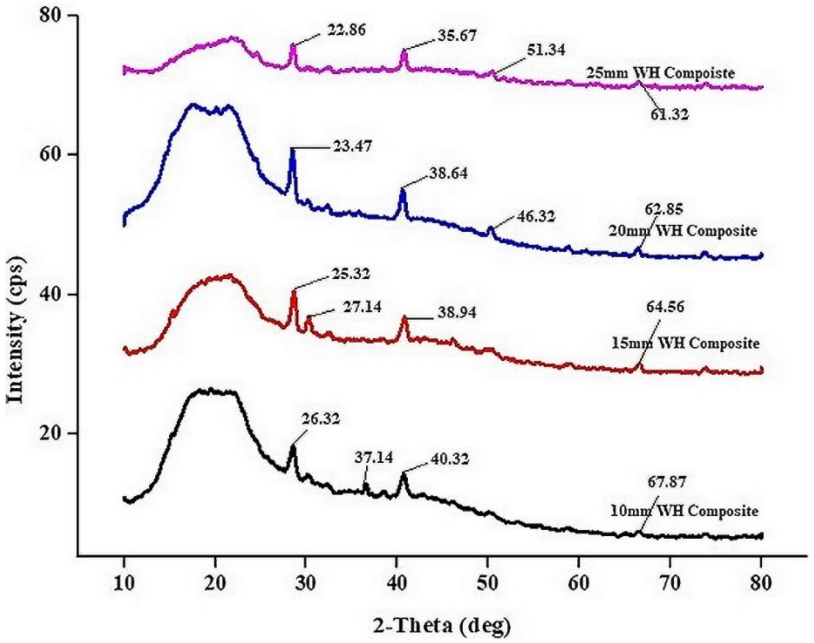

Figure 3 XRD spectra of WH composite (30 wt.%)

Table 1 Crystalline Peak, Lower Intensity Peak, Crystallinity Index of WH Polymer Composite

Serial No.	Area of Crystalline Peaks (A_c)	Area of All Peaks (A_a)	Crystallinity Index, CI (%)
1	435.12	899.76	48.41
2	467.14	913.76	51.12
3	492.76	812.2	60.68
4	455.80	801.27	56.27

6.3 FTIR Characterization

Fourier transforms analysis technique was used to investigate the chemical functional groups within the WH composites. Figure 4 explains the chemical functional groups of water hyacinth natural fibre reinforced polymer composite. The 10 mm WH composite band peak 3481.857cm^{-1} identified the oxygen-hydrogen (O-H) stretching of the cellulose and hemicellulose contents. In general, the peaks occurred within the range of 3360-3420 cm^{-1}. For the remaining composites with 15 mm, 20 mm, and 25 mm long fibres, the peaks occurred at 3419.07 cm^{-1}, 3425.480cm^{-1}, 3421.480cm^{-1} respectively. The carbon-hydrogen peaks occurred in the composites at 2883.9 cm^{-1},

2965.54 cm^{-1}, 2807.69 cm^{-1}, 2959.13cm^{-1}. This stretching showed that the vibration of total hydrocarbon molecules of the reinforcement phase originated from the cellulose and hemicellulose contents. Carbon, oxygen double bond (C=O) peaks represented the presence of hemicellulose in the sample. The carbon hydrogen double bond (C=H) peaks were identified at 1620.354cm^{-1}, 1686.472cm^{-1}, 1652.35cm^{-1}, 1643.21cm^{-1}. The peaks of the WH composites at 1517.62cm^{-1}, 1524.635cm^{-1}, 1562.327cm^{-1}, 1532.486cm^{-1} represented the C-C aromatic group and the lignin contents from the reinforcement area was indicated by the OCH3 groups. The carbon, hydrogen symmetrical determination and carbon-oxygen (C=O) double bond peaks were identified at 1236.51 cm^{-1}, 1237.962cm^{-1}, 1212.953cm^{-1}, 1235.147cm^{-1}. Carbon-oxygen single bond (C-O), carbon-carbon single bond (C-C), and (C-O)/(C-C) stage of peaks represented the hyacinth composites at 1030.562 cm^{-1},1236.55 cm^{-1}, 1045.72 cm^{-1}, 1053.926 cm^{-1}. Proper drying and removal of moisture decreased the lignin and hemicellulose contents because of the O-H bonding in the WH fibre phase. The peak at 1652.354 cm^{-1} in 20mm composite indicated an increase in the cellulose content of the particular composite because of proper moisture removal. The other peaks occurred for different length of the fibre composites and the raw peaks of their own is also presented in the chart.

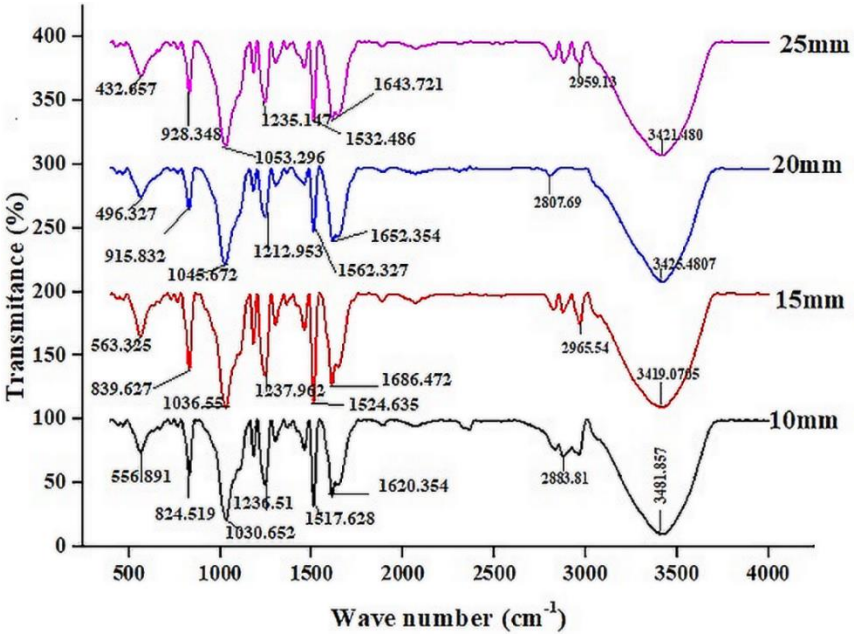

Figure 4 FITR Characterization of WH Composites

6.4 Fractured Surface Characteristics

The fractured surfaces of the WH fibre reinforced composites are placed in a hot air oven (64°C) for 24 hr to remove any moisture. Through this process, the wax substance and surface impurities were highly reduced. Figure 5 shows the primary reinforcement and secondary phase epoxy adhered well leading to good mechanical properties. Figure 5(a) shows the presence of minute impurities in the fibre phase in the WH composite surface. It was likely that this impurity resulted from the mechanical extraction process. Some minute dust particles were also present on the upper surfaces of the fibres possibly attached during the fibre processing. The high amount of fibre content created the agglomeration effect within the matrix phase. Figure 5(b) clearly demonstrated the fibre clusters leading to the poor interfacial bonding with the epoxy matrix, which eventually degraded the mechanical properties of the composite. Figure 5(c) shows that the failure of the composite sample under the impact load and fibres were pulled out from the matrix phase.

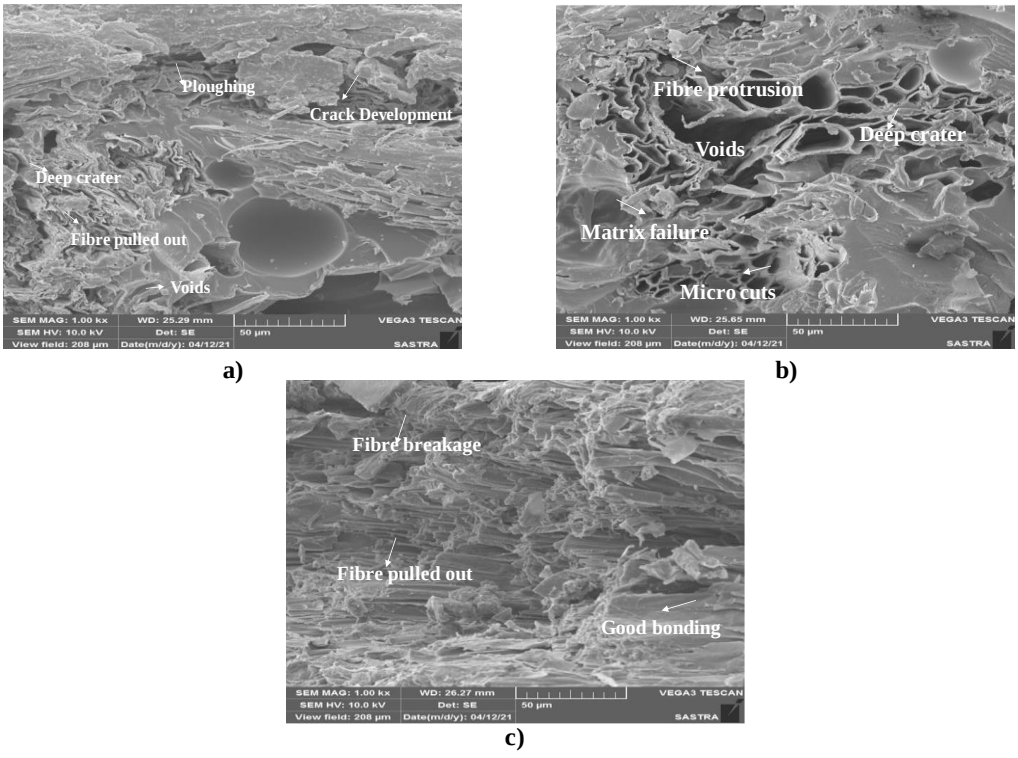

Figure 5 Fractured surface of the WH composites a) tensile failure b) flexural failure c) impact failure (20 mm fibre length and 30 wt.%)

7. ORGANISATION OF THESIS

The organization of the thesis is as below:

Chapter 1: This chapter introduces the natural fibre reinforced polymer composites for their mechanical properties, thermal properties, characterization studies. water resistance, and chemical resistance. A discussion was also held regarding the need and importance of adding egg shell particles as a filler material on the secondary reinforcement to the natural fibre reinforced composites.

Chapter 2: In this chapter an elaborated literature is reviewed based on the recent publications focused on water hyacinth natural fibres, mechanical properties of hyacinth fibre composites, powder and ash composites, thermal behaviour of water hyacinth fibre, powder and ash composites, characterization studies on hyacinth fibre, powder and ash reinforced composite materials, physical and characterization studies on eggshell filler materials are discussed.

Chapter 3: In this chapter covered the detailed information regarding the collection of water hyacinth plants, separation processes, different types of extraction process to extract the water hyacinth fibre from the parent plant stem, extraction process of hyacinth plant powder and ash particles from the dried hyacinth parent plant parts, eggshell filler material extraction processes, and the composite production (compression moulding) processes are discussed. The different types of testing's like mechanical testing (tensile, flexural, and impact), hardness tests, water and chemical absorption tests, thermal behaviour studies, characterization studies are briefly discussed.

Chapter 4: This chapter briefly discussed about the water hyacinth powder particle extraction from the parent water hyacinth plant, powder reinforced composite making processes, physical characterization of water hyacinth powder particles, water hyacinth powder composites mechanical testing's (tensile, flexural, and impact), hardness testing, characterization studies of hyacinth powder particles and different weight percentages of hyacinth

powder reinforced composite samples, water and chemical absorption studies based on hyacinth powder reinforced polymer composites, thermal behaviour of water hyacinth powder composites.

Chapter 5: This chapter detailed the water hyacinth ash particle conversion process from the dried water hyacinth parent plant parts, physical characterization of hyacinth ash particles. The mechanical testing like tensile, flexural and impact tests, hardness tests are taken to the hyacinth ash particle reinforced epoxy polymer composites.

Chapter 6: This chapter briefly discussed about the water hyacinth fibre extraction process from the parent plant of water hyacinth. The different types of extraction process are tried to extract the hyacinth fibre from the plant. The novel mechanical way of fibre extraction machine is designed to effectively extract the hyacinth fibre. The physical and chemical characterization, single fibre tensile strengths are taken to the extracted hyacinth fibre. The pure epoxy polymer matrix composite, hyacinth fibre reinforced polymer composites mechanical strength is tested.

Chapter 7: This chapter is briefly explained the preparation of eggshell filler materials, physical cauterization and different types of elements are presents in the eggshell filler materials are deeply investigated. The eggshell filler materials are added to the secondary matrix materials with different weight percentage ratios. Then, the eggshell filler material added hyacinth powder, ash, and fibre reinforced composite samples mechanical testing and hardness tests are done.

Chapter 8: Based on the result and discussions of the experimental investigation of hyacinth powder, ash, and fibre reinforced composite samples, eggshell filler added hyacinth powder, ash, and fibre reinforced composite samples detailed conclusion is reported.

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9. LIST OF PUBLICATIONS

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4. **A. Ajithram,** J. T. Winowlin Jappes, I. Siva and N. C. Brintha, Egg Shell Powder Extraction and its Application for Water Hyacinth (Eichhornia crassipes) Natural Fibre Polymer Composite Mechanical, Thermal behaviour and Characterization Analysis: Aquatic Waste into Sustainable Product, *Part L: Journal of Materials, Design and Applications*, (2021). DOI: https://doi.org/10.1177/14644207211064419

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